Waters
Lab Highlights

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LIQUID CRYSTALS
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ULTRAPURE LIQUID CRYSTALS AT THE KILOGRAM SCALE ON THE PREP 500A

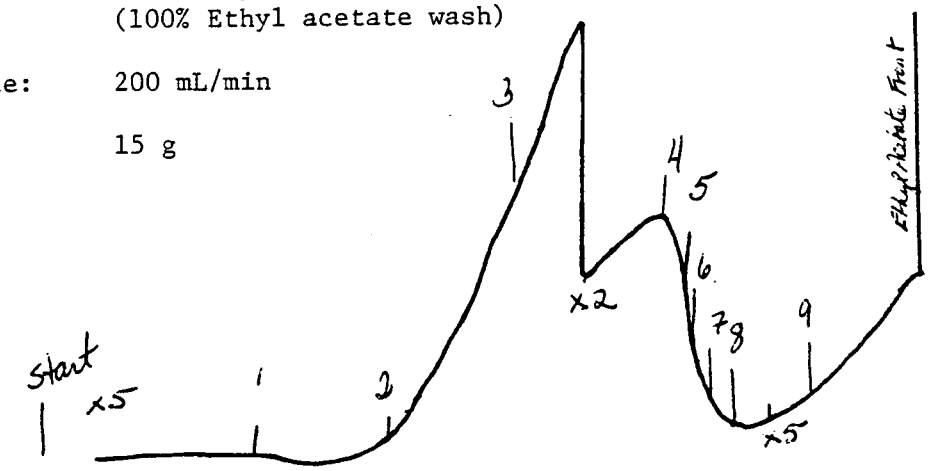
A customer wished to purify a contaminated batch of liquid crystals. The reverse-phase gradient separation of the mixture showed five major components with at least fifteen impurity peaks and the customer was interested in removing only one 0.22% impurity. However, sample solubility precluded the use of reverse-phase columns. Therefore, the following normal-phase conditions were developed using an analytical instrument.

Column: 2 PrepPAK® silica cartridges

Mobile Phase: 1.5% Ethyl acetate in hexane
(100% Ethyl acetate wash)

Flow Rate: 200 mL/min

Sample: 15 g



Working with the customer on a small scale, one gram separation, it was concluded that the 0.22% impurity appeared in the last fraction (Fraction 9). Recrystallization of this fraction effectively removed this impurity. Fifteen grams were then injected. Fractions 2-4 and 9 contained the desired material. Fractions 5-8 contained other impurities which the customer decided to discard. Fraction 9 was recrystallized two times in acetonitrile and then added to Fractions 2-4. The amount of the undesirable impurity was now down to 0.04%, an acceptable level.

Resolution of the separation was great enough to allow for loads of 20-25 grams and flow rates of 250-300 mL/min. Material from the second peak can be combined after each injection, recrystallized in bulk, and then added to the other clean fractions. With eight injections per day, 1 kilogram of material can be purified in a week.

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